PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference FP18983:MRP	FOR FURTHER ACTION	See Form PCT/IPEA/416
International application No. PCT/AU2004/000005	International filing date *(day/month/year)* 6 January 2004	Priority date *(day/month/year)* 7 January 2003
International Patent Classification (IPC) or national classification and IPC Int. Cl.[7] C01F 7/02, C09C 1/40, 1/68, 3/00, 3/04		
Applicant ADVANCED NANO TECHNOLOGIES PTY LTD et al		

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 3 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
   a. [X] *(sent to the applicant and to the International Bureau)* a total of 8 sheets, as follows:
      - [X] sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
      - [ ] sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.

   b. [ ] *(sent to the International Bureau only)* a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or table related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).
4. This report contains indications relating to the following items:

[X]	Box No. I	Basis of the report
[ ]	Box No. II	Priority
[ ]	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
[ ]	Box No. IV	Lack of unity of invention
[X]	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
[ ]	Box No. VI	Certain documents cited
[ ]	Box No. VII	Certain defects in the international application
[ ]	Box No. VIII	Certain observations on the international application

Date of submission of the demand 4 August 2004	Date of completion of the report 6 December 2004
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer ALBERT S. J. YONG Telephone No. (02) 6283 2160

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/AU2004/000005

Box No. I Basis of the report

1. With regard to the language, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ This report is based on translations from the original language into the following language which is the language of a translation furnished for the purposes of:

☐ international search (under Rules 12.3 and 23.1 (b))

☐ publication of the international application (under Rule 12.4)

☐ international preliminary examination (under Rules 55.2 and/or 55.3)

2. With regard to the elements of the international application, this report is based on (*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report*):

☐ the international application as originally filed/furnished

☒ the description:

pages 1-2,8-16, 21 (abstract) as originally filed/furnished
pages* 3-7 received by this Authority on 4 August 2004 with the letter of 4 August 2004
pages* received by this Authority on with the letter of

☒ the claims:

pages as originally filed/furnished
pages* as amended (together with any statement) under Article 19
pages* 17-19 received by this Authority on 4 August 2004 with the letter of 4 August 2004
pages* received by this Authority on with the letter of

☒ the drawings:

pages 1/4-4/4 as originally filed/furnished
pages* received by this Authority on with the letter of
pages* received by this Authority on with the letter of

☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing.

3. ☒ The amendments have resulted in the cancellation of:

☐ the description, pages
☒ the claims, Nos. 20-27
☐ the drawings, sheets/figs
☐ the sequence listing (*specify*):
☐ any table(s) related to the sequence listing (*specify*):

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

☐ the description, pages
☐ the claims, Nos.
☐ the drawings, sheets/figs
☐ the sequence listing (*specify*):
☐ any table(s) related to the sequence listing (*specify*):

* *If item 4 applies, some or all of those sheets may be marked "superseded."*

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/AU2004/000005

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims 1-19	YES
	Claims	NO
Inventive step (IS)	Claims 1-19	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-19	YES
	Claims	NO

2. Citations and explanations (Rule 70.7)

NOVELTY AND INVENTIVE STEP

Claims 1-19: The claimed invention relates to a process for the production of plate-like alumina particles comprising the steps of mixing nano-sized particles of an aluminium precursor compound and a diluent and heat treating the mixture below the melting point of the diluent.

None of the documents cited in the International Search Report discloses the requirement to conduct the heat treatment at below the melting point of the diluent. The closest art, US 6015456, discloses the heat treatment of a mixture of nano-sized aluminium hydroxide and ammonium phosphate (melting point = 190°C) at 600°C. Hence, the claims are novel and inventive in the light of the cited art.

starting materials, ie the salts, are completely dissolved as a first step. The process of *Merck* is preferably conducted at high temperatures between 900 and 1400°C.

The present invention was developed with a view to providing an alternative process for the production of substantially discrete plate-like alpha alumina particles with a high aspect ratio.

It will be clearly understood that, although prior art use and publications are referred to herein, this reference does not constitute an admission that any of these form a part of the common general knowledge in the art, in Australia or in any other country.

Throughout this specification and in the appended claims, the term "comprising" is used inclusively, in the sense that there may be other features and/or steps included in the invention not expressly defined or comprehended in the features or steps subsequently defined or described. What such other features and/or steps may include will be apparent from the specification read as a whole.

Summary of the Invention

According to a first aspect of the present invention there is provided a process for the production of plate-like alumina particles with a high aspect ratio, the process comprising the steps of:

forming a mixture of nano-sized particles of an aluminium precursor compound and a sufficient volume fraction of a diluent; and

heat treating the mixture to form substantially discrete plate-like alpha alumina particles dispersed in the diluent, characterised in that the step of heat treating the mixture is conducted below the melting point of the diluent.

The term nano-sized would be readily understood by persons skilled in the relevant art to refer to particles that are

smaller than one micron in size. The preferred size range for the nano-sized particles of the present invention is less than 100nm and preferably less than 50nm.

It is to be clearly understood that the product of the process need not comprise only alpha alumina particles. For example, gamma alumina particles may also be present. Moreover, the temperature for the step of heat treating may be selected to control the relative amounts of gamma alumina particles and plate-like alpha alumina particles that form. For example, if a preferred product requires that only 50% of the particles be in the form of plate-like alpha alumina, the step of heat treating may be conducted at a lower temperature than that selected to produce a product requiring 90% plate-like alpha alumina particles.

Preferably the nano-sized particles of the aluminium precursor compound are substantially discrete, ie there is close agreement between the average particle size determined using scanning electron microscopy and laser light scattering particle size analysis.

Preferably, the process further includes the step of removing the diluent from the substantially discrete plate-like alpha alumina particles after the step of heat treating.

The term "diluent" here is used to describe a substance in solid or liquid form that "dilutes" the mixture and is added to help maintain separation of the particles of both the precursor alumina compound and/or the plate-like alpha alumina particles throughout the process. The diluent may react with the aluminium precursor compound or be present as a spectator.

Preferably, the diluent is soluble in a solvent which selectively removes the diluent and does not react with the plate-like alpha alumina particles and the step of removing the diluent after the step of heat treating comprises the

step of washing with the solvent. More preferably the solvent is water or an alcohol.

Preferably, the sufficient volume fraction of the diluent is at least 80% of the total volume of the mixture.

While a wide range of diluents may be used, the preferred diluents are selected to encourage plate-like growth of the alpha alumina particles during heat treatment. A preferred diluent is a metal salt such as sodium sulphate, potassium sulphate or sodium chloride. Sodium chloride is highly preferred as being cheap and readily available.

A mineraliser in the form of a metal fluoride may also be added to the diluent to form a diluent-mineraliser system. The preferred metal fluorides are sodium fluoride, calcium fluoride, aluminium fluoride and sodium aluminium fluoride (cryolite).

The conditions for heat treatment of the mixture depend on the particular diluent or diluent-mineraliser system used. The advantage of using a diluent-mineraliser system is that the step of heat treating may be conducted at a lower temperature than for a diluent used alone.

Preferably, the step of heat treating the mixture is conducted below the diluent-mineraliser system in order to maintain separation between the plate-like particles as they form.

Preferably, the aluminium precursor compound is aluminium hydroxide. Aluminium hydroxide is cheap, readily available, easy to handle and readily dehydrates to form aluminium oxide.

The process described in WO 99/59754 and incorporated herein by reference may be used to produce substantially discrete nano-sized particles of an alumina precursor compound as the starting material for the process according

to the first aspect of the present invention.

Other suitable aluminium precursor compounds include aluminium sulphate, aluminium nitrate and aluminium chloride. With these precursor compounds, the process preferably further comprises the steps of milling the aluminium precursor compound with a suitable diluent such as sodium hydroxide in accordance with the method described in the applicant's International application WO 99/59754, the contents of which are incorporated herein by reference.

According to a second aspect of the present invention there is provided a process for the production of substantially discrete ultrafine plate-like alumina particles having a high aspect ratio, the process comprising:

milling a mixture of an aluminium precursor compound and a sufficient volume fraction of a diluent to form a dispersion of nano-sized particles of an intermediate aluminium compound in the diluent; and,

thereafter heat treating the dispersion to convert the nano-sized particles of the intermediate aluminium compound to substantially discrete plate-like particles of alpha alumina.

Preferably the process further comprises the step of removing the diluent such that ultra-fine plate-like particles are left behind in the form of an ultrafine powder. Preferably the step of removing the diluent includes the step of washing with a solvent which selectively dissolves the diluent while not reacting with the plate-like alumina particles.

The intermediate aluminium compound would typically be aluminium hydroxide or aluminium oxide. The precursor aluminium compound would typically be aluminium hydroxide.

According to a third aspect of the present invention there is provided a product in accordance with the processes described above. Such a product is suitable for use in the

following applications: soft focus cosmetics, pearlescent pigments, ceramic components, die coatings, and hard coatings.

The plate-like alpha alumina particles produced in accordance with the various aspects of the present invention having an aspect ratio of width to diameter of between 1:10 and 1:100 and more preferably between 1:20 and 1:50. The preferred aspect ratio depends on the particular application for which the powders are required. The plate-like alumina particles have a diameter between 0.1 to 30 microns. Preferably the plate-like alumina particles have a thickness of between 50 and 200 nm.

Brief Description of the Drawings

In order to facilitate a more detailed understanding of the nature of the invention, preferred embodiments of the process for the production of ultrafine plate-like alumina particles will now be described in detail, by way of example, only, with reference to the accompanying drawings, in which:

Figure 1 is a Scanning Electron Micrograph of alumina plate-like particles in accordance with Example 1;

Figure 2 is a Scanning Electron Micrograph of alumina plate-like particles, many of which are interlocked due to intergrowth made in accordance with Example 2;

Figure 3 is a Scanning Electron Micrograph of alumina plate-like particles made in accordance with Example 3;

Figure 4 is a Scanning Electron Micrograph of alumina plate-like particles made in accordance with Example 4;

Figure 5 is a graphical representation showing the size distribution of the diameter of the plate-like particles of Figure 4;

Figure 6 is a Scanning Electron Micrograph of alumina plate-like particles made in accordance with Example 5;

PCT/AU2004/000005
Received 04 August 2004

CLAIMS:

1. A process for the production of plate-like alumina particles comprising the steps of:

forming a mixture of nano-sized particles of an aluminium precursor compound and a sufficient volume fraction of a diluent; and

heat treating the mixture to form substantially discrete plate-like alpha alumina particles dispersed in the diluent, characterised in that the step of heat treating the mixture is conducted below the melting point of the diluent.

2. A process for the production of plate-like alumina particles according to claim 1 further comprising the step of removing the diluent after the step of heat treating.

3. A process for the production of plate-like alumina particles according to claim 2 wherein the diluent is soluble in a solvent and the step of removing the diluent from the mixture comprises the step of washing with the solvent after the step of heat treating.

4. A process for the production of plate-like alumina particles according to claim 3 wherein the solvent is water or an alcohol.

5. A process for the production of plate-like alumina particles according to any one of the preceding claims wherein the sufficient volume fraction of the diluent is at least 80% of the total volume of the mixture.

6. A process for the production of plate-like alumina particles according to any one of the preceding claims wherein the diluent is a metal salt.

7. A process for the production of plate-like alumina particles according to claim 6 wherein the metal salt is selected from the group comprising; sodium sulphate,

PCT/AU2004/000005
Received 04 August 2004

potassium sulphate and/or sodium chloride.

8. A process for the production of plate-like alumina particles according to any one of the preceding claims further comprising the step of adding a mineraliser to the diluent to form a diluent-mineraliser system.

9. A process for the production of plate-like alumina particles according to claim 8 wherein the mineraliser is a metal fluoride.

10. A process for the production of plate-like alumina particles according to claim 8 wherein the metal fluoride is selected from the group comprising further comprising; sodium fluoride, calcium fluoride, aluminium fluoride and/or sodium aluminium fluoride.

11. A process for the production of plate-like alumina particles according to any one of claims 8 to 10 wherein the step of heat treating the mixture is conducted below the liquidus of the diluent-mineraliser system.

12. A process for the production of plate-like alumina particles according to any one of the preceding claims wherein the aluminium precursor compound is selected from the group comprising; aluminium hydroxide, aluminium sulphate, aluminium nitrate and/or aluminium chloride.

13. A process for the production of plate-like alumina particles according to any one of preceding claims wherein the nano-sized particles of the alumina precursor compound are substantially discrete.

14. A process for the production of ultrafine plate-like alumina particles having a high aspect ratio comprising the steps of:

milling a mixture of an aluminium precursor compound and a sufficient volume fraction of a diluent to form a dispersion of nano-sized particles of an intermediate

PCT/AU2004/000005
Received 04 August 2004

aluminium compound in the diluent; and,

thereafter heat treating the dispersion to convert the nano-sized particles of the intermediate aluminium compound to substantially discrete plate-like particles of alpha alumina.

15. A process for the production of plate-like alumina particles according to claim 18 further comprising the step of removing the diluent from the mixture after the step of heat treating.

16. A process for the production of plate-like alumina particles according to claim 19 wherein the step of removing the diluent comprises the step of washing with a solvent which selectively dissolves the diluent while not reacting with the plate-like alumina particles.

17. A process for the production of plate-like alumina particles according to any one of claims 18 to 20 wherein the precursor aluminium compound is aluminium hydroxide or aluminium oxide.

18. A process for the production of plate-like alumina particles substantially as herein described with reference to and as illustrated in any one of the Examples 3 to 5.

19. A process for the production of plate-like alumina particles substantially as herein described with reference to and as illustrated in any one of the Figures 3 to 7.